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(21) International Application Number: PCT/EP98/01289 (22) International Filing Date: 2 March 1998 (02.03.98) (30) Priority Data: 9705346.6 14 March 1997 (14.03.97) GB (71) Applicant (for AU BB CA GB GH GM IE IL KE LC LK LS MN MW NZ SD SG SL SZ TT UG ZW only): UNILEVER PLC [GB/GB]; Unilever House, Blackfriars, London EC4P 4BQ (GB). (71) Applicant (for all designated States except AU BB CA GB GH GM IE IL KE LC LK LS MN MW NZ SD SG SL SZ TT UG US ZW): UNILEVER N.V. [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL). (72) Inventors; and (75) Inventors/Applicants (for US only): BARTOLETTI, Marcella, Margherita, Leda [IT/IT]; Unilever Italia S.p.A., Divisione Lever Industriale, Via Lever Gibbs, I-20071 Casalpusterlengo (IT). MORRIS, Ronald, Meredith [GB/IT]; Unilever Italia S.p.A., Divisione Lever Industriale, Via Lever Gibbs, I-20071 Casalpusterlengo (IT). TUMMIOLO, Roberto [IT/IT]; Unilever Italia S.p.A., Divisione Lever Industriale, Via Lever Gibbs, I-20071 Casalpusterlengo (IT).		(74) Agent: TANSLEY, Sally, Elizabeth; Unilever PLC, Patent Dept., Colworth House, Sharnbrook, Bedford MK44 1LQ (GB). (81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
(54) Title: LAVATORY CLEANSING COMPOSITIONS		
(57) Abstract		
<p>A slow-release lavatory cleansing block comprising: a) a bleaching agent or precursor therefor, b) an oily liquid perfume, and c) a water-insoluble, gelling polymer derived from one or more ethylenically unsaturated carboxyl group-containing monomers.</p>		

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- 1 -

LAVATORY CLEANSING COMPOSITIONS5 Technical Field

This invention is concerned with improvements in and relating to lavatory cleansing compositions. More particularly, the invention is concerned with solid lavatory
10 cleansing compositions which may be used to impart cleansing and/or other components to the flush water of a lavatory or urinal by placement of the composition in the cistern of the lavatory or urinal.

15

Background to the Invention

The use of solid slow-release compositions to impart cleansing and/or other components to the flush water of a
20 lavatory or urinal is well known. Such compositions may, for example, be immersed in the water of a lavatory cistern either in free-standing form or in containerised form (i.e. in a dispensing container which allows for release of components of the solid composition in solution in water in
25 a more or less metered fashion, on flushing of the cistern).

Alternatively, the solid composition may be held under the rim of a toilet, in a suitable holder, for intermittent contact of the solid material with flush water.

30 For convenience these two approaches will be referred to as 'cistern-blocks' and 'rim-blocks' herein. Such blocks produce foam, often produce a pleasing odour and can have germicidal properties.

- 2 -

Solid lavatory cleansing compositions typically comprise a surfactant component, generally together with one or more fillers or builders (e.g. inorganic salts such as sodium sulphate, sodium chloride etc.). Commonly, such

5 compositions also contain a dyestuff and, frequently, a dissolution retardant material. GB1364459 and GB1364460 disclose that the incorporation of a hydrophobic material such as a wax, stearin, long chain alcohol (up to 6EO), paradichlorobenzene or ethylene glycol monostearate in a

10 lavatory block enables the solubility rate of the block to be controlled.

EP0167210 is concerned with a lavatory rim-block which is free of the solid perfume component para-dichlorobenzene

15 (PDCB). The block contains as essential ingredients anionic surfactant (40-90%wt) an inert or electrolyte filler (5-55%wt) and an oily liquid perfume (5-55%wt). The block may also contain non-surfactant nonionic polymeric materials such as polyethylene glycols and minor ingredients such as

20 dyes, germicides, fungicides, bleaches, opacifiers and the like.

EP 0 073 542 describes a lavatory cleansing block comprising a partially esterified copolymer of vinyl methyl ether and

25 maleic anhydride.

It is also known to use polymers based on ethylenically unsaturated carboxyl group-containing monomers, such as polyacrylates, in solid lavatory cleansing compositions.

30 Such compositions having mineralisation prevention properties is disclosed in US4861511. The compositions disclosed therein contains from 2% to 15% by weight of a polyacrylate having a molecular weight in the range 500 - 50,000. As used herein, any reference to an average

- 3 -

molecular weight means the weight average molecular weight, unless specifically recited to the contrary.

Higher average molecular weight water-soluble materials are
5 used in WO96/28536, which relates to a slow release lavatory
block which comprises a water soluble polymer derived from
one or more ethylenically unsaturated carboxyl group-
containing monomers and having an average molecular weight
greater than 50,000. Polymers of acrylic acid with
10 molecular weights of about 90,000 (such as Degapas 4104N)
are preferred.

The dissolution retardant is of particular importance when
the solid composition is to be formulated as a cistern-
15 block. These problems are compounded when a halogen release
agent or other bleaching agent is present. From EP0206725
it is clear that halogen release agents are, by their
nature, powerful chemically reactive species, serving as
halogenating or oxidising agents. Thus, in practice, it is
20 found that halogen release agents tend to react with surface
active materials and/or tend, when moistened, to evolve gas
destroying the physical integrity of the cleaning
composition, particularly in the case of free standing
blocks for immersion in the cistern of a lavatory. These
25 processes make it difficult to formulate a block which
achieves a reasonable in use lifetime, i.e. around 4-6 weeks
when the toilet is flushed some 15 times per day.

30 Brief Description of the Invention

We have determined that improved in-cistern blocks can be
formulated which comprise: a bleaching agent (or its
precursor), an oily liquid perfume and a water-insoluble

- 4 -

polymer derived from one or more ethylenically unsaturated carboxyl group-containing monomers.

Accordingly the present invention provides a slow-release
5 lavatory cleansing block comprising:

a) a bleaching agent or precursor therefor,

b) an oily liquid perfume, and,

10

c) a water-insoluble gelling polymer derived from one or more ethylenically unsaturated carboxyl group-containing monomers.

15 It is believed that the combined use of these materials enables the production of a bleaching in-cistern block which has an acceptable in-use lifetime due to improved stability. Without wishing to restrict the invention by reference to
any theory of operation it is believed that the polymer
20 forms a protective layer at the surface of the block which retards the access of water to the block and prevents rapid decomposition of the block.

25 Detailed Description of the Invention

In order that the invention may be further understood the following description details preferred and optional
features of the invention.

30

Polymers

The water-insoluble, gelling polymers used in the
35 composition of the invention are polycarboxylic acids

- 5 -

derived from one or more ethylenically unsaturated carboxyl group-containing monomers, especially ethylenically unsaturated carboxylic acids such as acrylic acid or maleic acid.

5

The carboxyl group-containing monomers may be polymerized alone or in combination with other ethylenically unsaturated monomers. The preferred polymers in embodiments of the present invention are those which are readily available in the marketplace. These are polymers of acrylic or methacrylic acid or maleic anhydride, or a co-polymer of one or more of the same either together or with other monomers. In general, polymers derived largely or wholly from the acidic monomers are preferred.

15

Suitable polymers include polyacrylic acid, polymaleic anhydride and copolymers of either of the aforementioned with ethylene, styrene and methyl vinyl ether.

20

Typical polymers are polyacrylic acid and acrylic acid/maleic acid copolymers. In practice the polymers are employed in the form of acids, but can also be employed as salts e.g. alkali metal salts such as the sodium salt. The use of the polymer salts is advantageous if it is required to reduce dust production during formulation and manufacture.

25

It is preferred that the polymer is cross-linked. The cross-linked polyacrylate polymers of the present invention are generally characterised as resins in the form of acrylic acid polymers. Such materials are available from a number of sources including materials available under the tradename CARBOPOL (TM) from B. F. Goodrich Company, the tradename SOKOLAN (TM) from the BASF Corporation and under the tradename POLYGEL (TM) from 3V Sigma. The cross-linked

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- 6 -

polyacrylate polymers are generally characterised as acrylic acid polymers which are cross-linked with an additional monomer or monomers in order to exhibit an effective molecular weight of one to seven million g/mole. The average formula weight for a polymer sub-unit is preferably of the order of 60-120 g/mole.

The polymer is suitably present in an amount of from 0.5 to 20% by weight, more preferably from 1 to 5% by weight, most preferably around 2-3% by weight thereof. Polygel DB (TM) ex 3V Sigma, a cross-linked high molecular weight polyacrylate, has been found to be a suitable material at an inclusion level of around 2-3%wt.

15

Surfactants

Preferably, the composition will also contain a surfactant component which may be anionic or nonionic in nature. The surfactant serves to provide a cleansing and foaming effect and may, also act synergistically with the polymer component. Typically, surfactant comprises up to 50%wt of the composition.

Preferably, the surfactant component comprises one or more anionic surface active agents, optionally in combination with one or more nonionic surface active agents. Suitable anionic surface active agents include alkali metal or ammonium alkylaryl sulphonates (especially alkyl benzene sulphonates), alkane sulphonates, alkyl sulphates and sarcosinates.

We have determined that improved foaming properties are obtained by the use of a surfactant system which comprises primary alkyl sulphate (PAS) together with other anionic

- 7 -

surfactants. Preferably the present invention provides a lavatory block comprising 1-15%wt (more preferably 1-5%wt) of a primary alkyl sulphate and 15-50%wt (more preferably 30-50%wt) of other anionic surfactants. We have determined
5 that the use of this mixed surfactant system is advantageous in that it reduces the stickiness of the block during manufacture. The blocks with 1-5% PAS show improved wear characteristics.

10 Preferably said other anionic surfactants is a sulphonate. Suitable sulphonates include alkyl benzene sulphonate (ABS). It is believed that the combination of relatively low levels of PAS together with higher levels of ABS promotes the foaming and the perfume delivery from the block. PAS is also
15 believed to be environmentally more acceptable than alkyl benzene sulphonate.

Suitable nonionic surfactants include polyethoxylated fatty alcohols, polyethoxylated fatty acids, polyethoxylated alkyl
20 phenols, amine oxides and ethylene oxide/propylene oxide block copolymers.

The total amount of surfactant when present, may lie within wide limits. In practice, the surfactant will generally for
25 10 to 70% by weight of the composition, but more preferable that surfactant comprises from 20 to 50% by weight thereof.

Process Aids

30 As will be described in further detail below extrusion is the preferred method of manufacture. Optionally, the blocks according to the invention further comprise a processing aid to assist in extrusion. Suitable processing aids include
35 oils (including both mineral and silicone oils), esters

- 8 -

(other than those derived from ethylenically unsaturated carboxyl group-containing monomers) and polybutene.

One particularly suitable processing aid is an alkoxyated alcohol. It is preferred that the alkoxyated alcohol is an ethoxyated alcohol. The preferred level of alkoxyated alcohol is 0.75-2%wt. The preferred ethoxy chain length is 40-60 with an average ethoxy chain length of around 50 being preferred. Suitable materials include Empilan KM 50/KF (TM) ex. Albright & Wilson. Another suitable process aid is a PEG monostearate. PEG 4000 Monostearate (100%) ex DAC is a suitable raw material. It is believed that an effect of the process aid is to make the block harder and easier to cut from an extruded billet.

15

Fillers

Typically, blocks according to the present invention comprise 0 to 50% by weight of an inert non-polymeric and/or electrolyte filler. Preferably blocks contain 5-50%wt, more preferably 10-30% of filler.

Suitable fillers include one or more of urea, sodium, magnesium and calcium carbonates, sodium chloride, borax, talc and sodium, magnesium and calcium sulphates. Preferred ionic fillers include sodium sulphate. Preferred inert, non-polymeric fillers include calcium carbonate.

Typical levels of total filler range from 10-40%wt on product in total.

It has been found useful, for ease of formulation and to ensure complete solubility of in-cistern blocks, to employ 10-30%wt of an ionic filler as the sole filler present.

- 9 -

For mixed filler systems, preferred levels of filler are 10-20%wt on product of ionic filler and 10-20% on product of inert filler. It is particularly preferred to use an approximately 50:50 mixture of sodium sulphate and calcium carbonate as the filler. For mixed filler systems typical levels in product are 15%wt of each of calcium carbonate and sodium sulphate.

10

Bleaching Agents

Typically the blocks comprise up to 50% by weight of an at least sparingly water soluble bleaching agent. Typical levels of bleaching agents are 2-30%wt on product. For the purposes of the present specification the term bleaching agent is used to mean both a bleaching agent and a precursor which produces a bleaching agent unless the context demands otherwise.

20

Suitable bleaching agents active-halide and active-oxygen bleaching agents, particularly the so-called 'halogen release agents'.

25

Chlorine bleaching agents are preferred. Suitable water-soluble, active chlorine, bleaching agents used in accordance with the invention include chlorinated cyanurates, phthalimides, p-toluene sulphonamides, azodicarbonamides, hydantoins, glycoluracils, amines and melamines. The alkali metal salts of cyanurates are preferred.

30

A particularly preferred bleaching agent is sodium dichlorocyanurate (NaDCCA). The bleaching agent is typically present in an amount of 10-30% and most preferably

35

- 10 -

at around 25%. Oxidan DCN/WSG (TM) ex Sigma has been found to be a suitable bleaching agent.

5 Perfume

Preferably, the blocks comprise 2-15%wt of a hydrophobic oily liquid perfume. The blocks more preferably comprise, 2-10%, more preferably 3-6%wt of the perfume. Levels of
10 around 4%wt perfume are particularly preferred. This oily perfume is typically of the kind described in the European patent application EP 167,210. It will be understood that the liquid oily perfume must be stable in the presence of the water-soluble, active chlorine, bleaching agent.
15 Suitable oily perfumes can be easily selected by testing them in combination with the water-soluble, active chlorine, bleaching agent.

Examples of suitable bleach-stable perfumes are Verdeo 898,
20 Bonanza 048 and Ponderosa 431 all ex IFF, and LB 132 ex Quest. Particularly preferred perfumes are Icebreaker Super Mod, Oxygen Supra Mod, Motebianco Supra and lemonfit Supra (all TM) ex Givaudan Roure. The most preferred perfume is Green Tank Harder (TM) ex. Givaudan Roure.

25

Minors

Minor components will generally be present but are optional.
30 These include colouring agents, and/or whiteners. These materials should be chosen such that they are compatible with the bleaching agent and do not react therewith to a significant extent. We have determined that 'Sudangelb 150' (TM) ex. Sandoz is an acceptable colouring agent as are
35 Colanyl Green (TM) ex Hoechst and Dispers Blue (TM) ex BASF.

- 11 -

A particularly preferred colouring agent is P. Green (CI 74260). Titanium dioxide is an acceptable whitener. Levels of colouring agents and/or whiteners are typically below 5%wt. For colouring agents levels are typically in the range
5 0.0001-0.1%wt.

Further enhancement of the product may be obtained by the additional use of chelating agent, sequestrant or water-softening agent such as ethylene diamine tetra-acetic acid
10 or a derivative thereof, nitrolotri-acetic acid, phosphonates of polyphosphates, metasilicates, boroheptonates, s.s-ethylene-diamino disuccinate, dipicolinic acid, 2-phosphonobutane-1,2,4-tricarboxylic acid, or lower molecular weight polymeric materials capable of inhibiting crystal
15 growth. Further reducing agents, such as alkali metal metabisulphates may be present to assist in the reduction of staining due to metals such as iron.

An optional minor component is a foam-boosting surfactant.
20 Suitable surfactants include amine oxides.

Process

25 Compositions in accordance with the invention may be produced by a variety of routes. For example, they may be prepared by a so-called "hot-melt" process comprising melting the fusible constituents of the block either alone and then adding other components in admixture with non-
30 fusible components, and subsequently casting the melt into moulds. More preferably, however, compositions in accordance with the invention are formed into the desired final shape by a compression technique, i.e. a technique involving the steps of forming a mixture of the ingredients of the

- 12 -

composition and then compressing that mixture into the desired shape.

An especially preferred process is an extrusion process in which the mixture of the components is extruded into a solid bar or rod which is subsequently cut into pieces of the desired size. In this connection, it may be noted that when the compositions of the invention are used as free-standing lavatory cleansing blocks, these suitably have a weight from 30 to 150gms. When extruding a solid composition it is generally advantageous, as noted above, that some lubricant component or process aid be present to facilitate extrusion.

Compositions in accordance with the invention may also be formed into the final desired shape by a tableting technique.

As used herein the term block is not intended to limit the shape of the eventual product. For cistern blocks the rod is cut into lengths which are short relative to their diameter.

Preferred Compositions

Preferred embodiments of the invention provide a slow-release lavatory cleansing block comprising:

- a) 2-30%wt of a halogen release agent,
- b) 2-15% wt of an oily liquid perfume, and,
- c) 1-5%wt of a water-insoluble, gelling polymer derived from one or more ethylenically unsaturated carboxyl group-containing monomers.

- 13 -

Particularly preferred compositions are:

- 5 a) 1-5%wt primary alkyl sulphate
 - b) 30-50%wt alkyl benzene sulphonate
 - c) 10-30%wt NaDCCA
 - 10 d) 10-30%wt sodium sulphate, calcium carbonate or a mixture thereof,
 - e) 2-10%wt oily liquid perfume
 - 15 f) 1-5%wt water insoluble, gelling, cross linked polyacrylate,
 - g) 0-5%wt colour and/or optical brightener
- 20 The invention also provides a method of cleaning a lavatory or urinal using a block of a composition in accordance with the invention.

In order that the invention may well be understood, the
25 following Examples are given by way of illustration only.

- 14 -

Examples

Blocks having the compositions A, B, C and D listed in Table 1 below were made up by an extrusion process. All amounts
5 are expressed as percentages by weights. The materials employed were:

	Alkyl benzene sulphonate	NANSA HS 80/LPF [TM] ex. Albright & Wilson
10	Primary alkyl sulphate	EMPICOL LZ-V [TM] ex. Albright & Wilson
15	Polymer	Polygel DB [TM] high molecular weight, water-insoluble, partially cross-linked acrylic acid polymer ex. Sigma.
20	NaDCCA	OXIDAN DCN/WSG [TM] ex Sigma
	Perfume	Green Tank Harder [TM] ex Givaudan Roure.
25	Colour	P. Green CI 74260 available as Colanyl Green GG 130 [TM] ex. Hoechst.

- 15 -

TABLE 1

Example	A	B	C	D
Alkyl benzene sulphonate	37%	37%	38%	33.5%
Primary alkyl sulphate	2.5%	4.5%	3.5%	8.0%
Polygel DB	2.0%	3.0%	2.0%	3.0%
NaDCCA	25%	25%	25%	25%
Sodium Sulphate	19.8%	16.7%	17.5%	17.2%
Perfume	4.0%	4.0%	4.0%	4.0%
Colour	0.02%	0.02%	0.02%	0.02%
Water	-----to100%-----			

5

All of the blocks gave satisfactory performance.

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- 16 -

CLAIMS

- 5 1. A slow-release lavatory cleansing block comprising:
- a) a bleaching agent or precursor therefor,
- b) an oily liquid perfume, and,
- 10 c) a water-insoluble, gelling polymer derived from one or more ethylenically unsaturated carboxyl group-containing monomers.
- 15 2. A block according to claim 1, wherein the polymer is selected from polyacrylic acid and acrylic acid/maleic acid copolymers, and metal salts thereof.
3. A block according to either preceding claim, wherein
- 20 the polymer is at least partially cross-linked.
4. A block according to any preceding claim, comprising from 0.5 to 20% by weight of the polymer.
- 25 5. A block according to any preceding claim, comprising 1 to 5% by weight of the polymer.
6. A block according to any preceding claim, further comprising a processing aid selected from mineral oil,
- 30 silicone oil, water insoluble esters other than those derived from ethylenically unsaturated carboxyl group-containing monomers , polybutene and alkoxyated alcohols.

- 17 -

7. A block according to any preceding claim wherein the bleaching agent is selected from chlorinated cyanurates, phthalimides, p-toluene sulphonamides, azodicarbonamides, hydantoins, glycoluracils, amines and melamines.
8. A block according to any preceding claim comprising:
- a) 2-30%wt of a halogen release agent,
 - b) 2-15% wt of an oily liquid perfume, and,
 - c) 1-5%wt of a water-insoluble, gelling polymer derived from one or more ethylenically unsaturated carboxyl group-containing monomers.
9. A block according to any preceding claim comprising:
- a) 1-5%wt primary alkyl sulphate
 - b) 30-50%wt alkyl benzene sulphonate
 - c) 10-30%wt NaDCCA
 - d) 10-30%wt sodium sulphate, calcium carbonate or a mixture thereof,
 - e) 2-10%wt oily liquid perfume
 - f) 1-5%wt water insoluble, gelling, cross linked polyacrylate,
 - g) 0-5%wt colour and/or optical brightener

INTERNATIONAL SEARCH REPORT

International Application No
PCT/EP 98/01289

A. CLASSIFICATION OF SUBJECT MATTER <div style="margin-left: 40px;">C 11 D 17/06</div>		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) <div style="margin-left: 40px;">C 11 D, E 03 D</div>		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practical, search terms used)		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	GB 2273106 A (JEYES GROUP PLC) 08 June 1994 (08.06.94), claims.	1, 7, 8, 9
A	DE 2750528 A1 (THE PROCTER & GAMBLE CO.) 24 May 1978 (24.05.78), example 1, claims 1, 2, 6, 13.	1, 8, 9
A	EP 0184416 A2 (JEYES GROUP LTD) 11 June 1986 (11.06.86), claims 1-3, 5, 9.	1, 8, 9
A	EP 0014979 A1 (HENKEL KGAA) 03 September 1980 (03.09.80), claims 1-3, 5, 9.	1, 8, 9
<div style="display: flex; justify-content: space-between;"> <input checked="" type="checkbox"/> Further documents are listed in the continuation of box C. <input type="checkbox"/> Patent family members are listed in annex. </div>		
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Date of the actual completion of the international search <div style="text-align: center;">22 June 1998</div>		Date of mailing of the international search report <div style="text-align: center; font-weight: bold;">20 JUL 1998</div>
Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+ 31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+ 31-70) 340-3016		Authorized officer <div style="text-align: center; margin-top: 10px;">SEIRAFI e.h.</div>

INTERNATIONAL SEARCH REPORT

-2-

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p style="text-align: center;">--</p> GB 1538857 A (CIBA-GEIGY) 24 January 1979 (24.01.79), the whole document. <p style="text-align: center;">----</p>	1,8,9

ANHANG

zum internationalen Recherchen-
bericht über die internationale
Patentanmeldung Nr.

ANNEX

to the International Search
Report to the International Patent
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ANNEXE

au rapport de recherche inter-
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PCT/EP 98/01289 SAE 189501

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